# FRACTURE TOUGHNESS AND MECHANICAL PROPERTIES OF PURE NIOBIUM AND WELDED JOINTS FOR SUPERCONDUCTING CAVITIES AT 4 K

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*Abstract*

The joint project for high-intensity proton accelerators in KEK and JAERI has been proposed with the superconducting proton linac. Because of the reduced-β (v/c, Particle velocity relative to the speed of light) and squeezed cavity shape, mechanical properties of cavities are very important. In this paper, the results of fracture toughness and mechanical tests of pure niobium (RRR≥200) plates (3-mm-thick)  and welded joints for superconducting cavities at 4 K are reported.

## 1  INTRODUCTION

Several mechanical properties of pure niobium (Nb) at low temperatures have been investigated [1] [2] [3]. But, fracture toughness data that enable the quantitative evaluation of the fracture behavior under the presence of a flaw are not available. In this paper, detailed results of fracture toughness tests at the liquid helium temperature of 4 K and fractographic/microscopic analysis are described, including the summary of the basic tensile and impact test results.

## 2  MATERIALS

The commercially available pure Nb plates with high RRR of over 200 for superconducting cavities were supplied by TOKYO DENKAI (Lot 1:Sep. 30, 1996, Lot 2:July 29, 1998) and prepared for the tests. Materials were cold-rolled and annealed at 750 °C for 2 hours at the vender. Test specimens were wire cut from as received materials and  joints were welded by using an Electron Beam Welding (EBW) machine at the KEK workshop. The thickness of the materials is 3 mm and a weld bead width of the joint is about 5 mm. Table 1 shows the chemical composition of the  material.

Table 1: Chemical composition of the material

| Element | H | O | N | C | Ta | Fe |
|---|---|---|---|---|---|---|
| Wt % | 0.001max | 0.01 | 0.004 | 0.004 | 0.095 | 0.003 |

| | Ti | W | Si | Mo | Zr | Nb |
|---|---|---|---|---|---|---|
| | 0.003 | 0.01max | 0.002 | 0.005max | 0.01max | 99.85min |

## 3  EXPERIMENTAL

### 3.1 Tensile and Impact Tests

The mechanical properties, 0.2% yield strength (YS), ultimate tensile strength (TS) and elongation were measured at 4 K, 77 K and room temperature (RT). Figure 1 and figure 2 summarize the tensile properties of two samples tested.
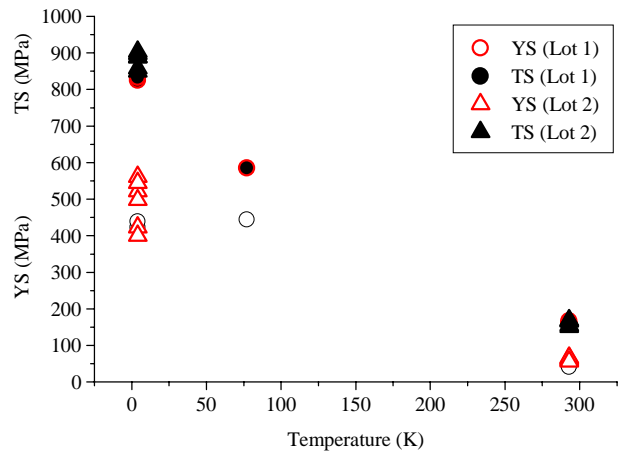


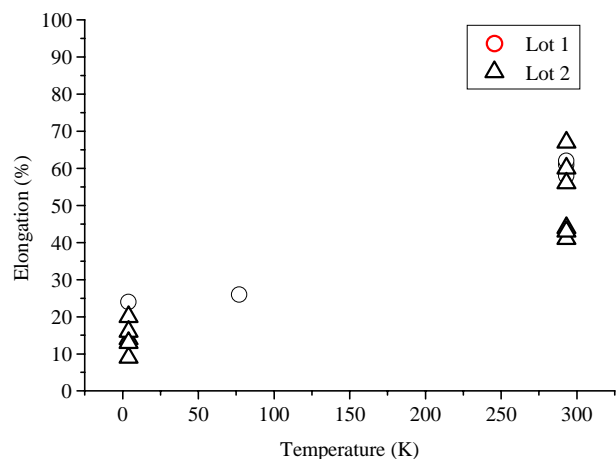Figure 1: Summary of yield and tensile strength data



Figure 2: Summary of elongation data

The results of V-notch Charpy impact tests at 4 K, 77 K and RT are summarized in figure 3. The thickness of test specimens is 3 mm.
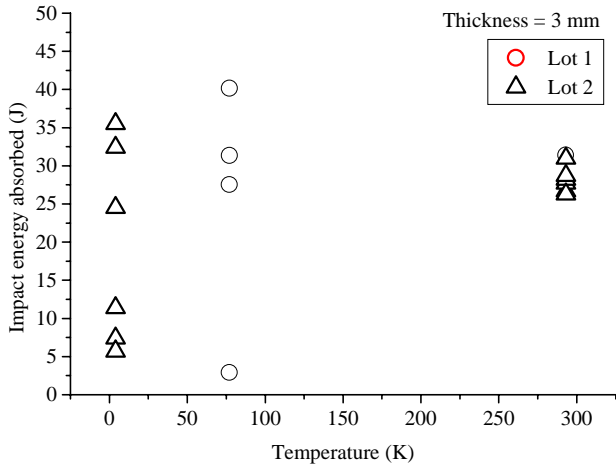


Figure 3: Summary of V-notch Charpy impact tests

## 3.2 Fracture Toughness Test

Plane-strain fracture toughness tests of Lot 1 material were performed according to the ASTM E399-90 [4]. Figure 4 shows the photograph of the test. In order for a result to be considered valid to this method, it is required to satisfy the specifications; a size of specimen, crack length, fatigue pre-cracking condition, etc . The Appendix summarizes the various specifications according to the ASTM E399, E1737 [5] and BS7448 [6] with the descriptions of detailed equations and symbols. As shown in figure 5, the shape of the test piece was made to be compact tension (CT) specimens of the half size, and the thickness was made to be 3 mm as of original thickness. In making the plane-strain fracture toughness tests valid, the biggest problem was an introduction of the fatigue pre-crack. The yield stress of this material at RT is 44 MPa. This means that the crack must be introduced in the very low stress intensity at RT in order to satisfy Eq. 1, and the very long term, which is unrealistic, is needed.

$$K_{f(max)} \leq 0.6 \, (\sigma_{y1}/\sigma_{y2}) \times K_Q \qquad (1)$$

The maximum stress intensity factors $K_{f(max)}$ for this present test assuming $K_Q=30$ MPa·m$^{1/2}$ at 4 K are 1.8 MPa·m$^{1/2}$ at RT and 19.1 MPa·m$^{1/2}$ at the liquid nitrogen (LN$_2$) temperature of 77 K. Therefore, the introduction of the pre-crack was carried out at the LN$_2$ temperature with $K_{f(max)}$ of $12.5 \sim 13.5$ MPa·m$^{1/2}$.

Figure 6 and figure 7 show the load – displacement curves of sample No.2 (Bulk Nb) and No.3 (Weld bead) obtained in the fracture toughness test. In case of the sample No.1, it seemed the excessive compressive load caused the plastic deformation by the error operation. The result of this sample is needed to be treated carefully.

On the basis of plane-strain criteria of Eq. 2, these results are revealed not valid, and applicable only for 3-mm-thick Nb materials.

$$B \, \& \, a \quad \geq \quad 2.5 \left( \frac{K_{IC}}{\sigma_{ys}} \right)^2 \qquad (2)$$

For example, in the case of sample No.2, a 28-mm-thick plate is required for plane-strain conditions to apply.

Table 2 summarizes the measured fracture toughness ($K_Q$), 95 % secant load ($P_Q$) and crack length ($a$). Because the plain-stress evaluation was needed, the $J_c$ calculation was made according to the ASTM E1737 [5] and BS7448 [6]. Table 3 shows the plane-stress fracture toughness ($J_c$) and converted toughness ($K_c$). Estimated $K_c$ values are much greater than the $K_Q$ values.
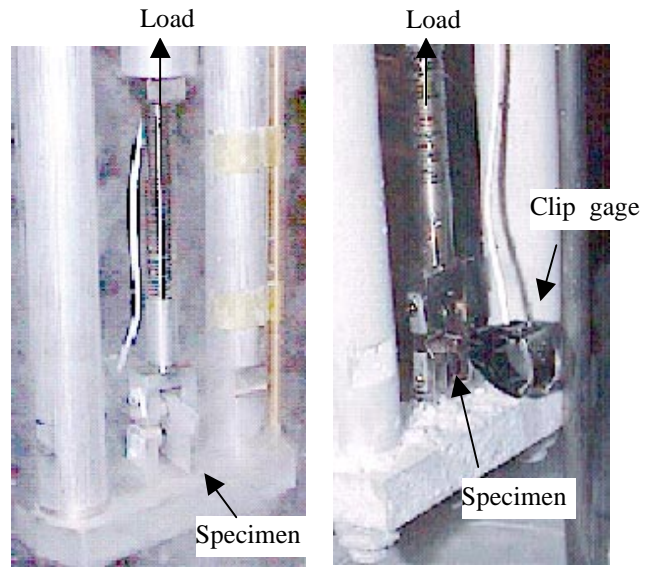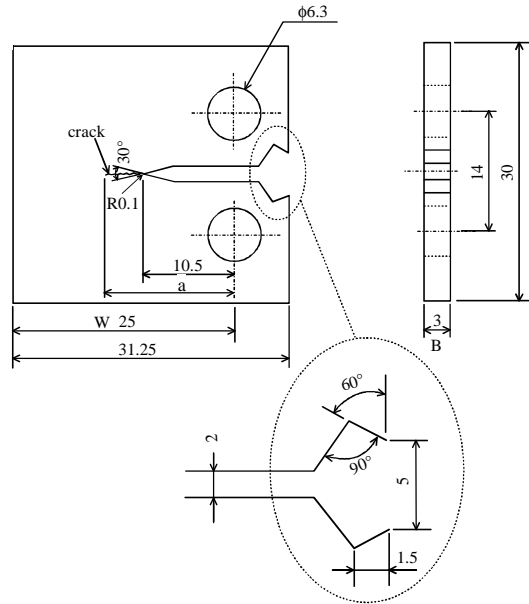


Figure 4: Fracture toughness test at 4 K



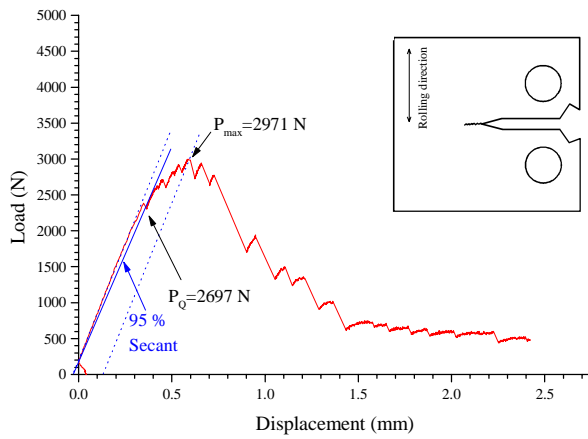Figure 5: Shape of fracture toughness specimen (1/2 CT)

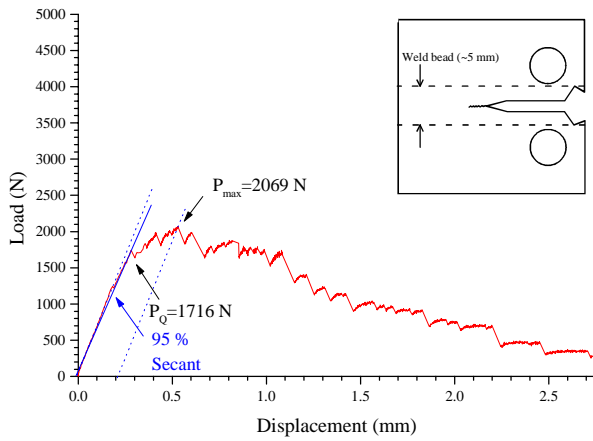Figure 6: Load – displacement curve (No.2, Bulk Nb)



Figure 7: Load – displacement curve (No.3, Weld bead)

Table 2: Fracture toughness $K_Q$ (for 3 mm thickness)

| No. | Condition | $K_Q$ | Load $P_Q$ | Crack length a |
|-----|-----------|-------|------------|----------------|
|     |           | MPa•m$^{1/2}$ | kN | cm |
| 1 | Nb TL | (50.1) | 2.680 | 1.206 |
| 2 | Nb LT | 45.5 | 2.370 | 1.228 |
| 3 | Weld bead | 33.5 | 1.720 | 1.240 |
| 4 | Weld bond | 35.0 | 1.765 | 1.256 |

Table 3: Plane-stress fracture toughness $J_c$

| No. | Condition | $J_c$ | Load $P_{max}$ | $K_c$ (from $J_c$) |
|-----|-----------|-------|----------------|--------------------|
|     |           | kJ/m$^2$ | kN | MPa•m$^{1/2}$ |
| 1 | Nb TL | (81.9) | 4.335 | (99.2) |
| 2 | Nb LT | 57.0 | 2.971 | 82.8 |
| 3 | Weld bead | 40.3 | 2.069 | 69.6 |
| 4 | Weld bond | 54.9 | 2.765 | 81.3 |

## 3.3 Fractographic and metallographic analysis

Correlations between fracture and microstructure/fracture surface were investigated by using metallographic and fractographic methods. Figure 8 shows the macroscopic fracture surfaces of samples No.1 to No.4. Figure 9 to figure 13 show the magnified fracture surfaces of the sample No.2 and No.3 by using a scanning electron microscope (SEM). These fractographic analyses indicate brittle and intergranular fracture mechanisms with the cleavage fracture surfaces.
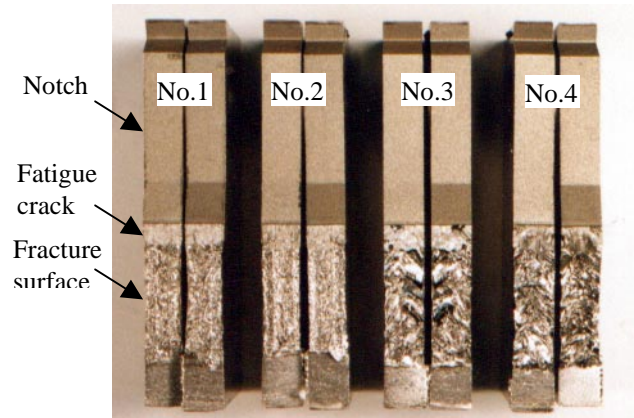


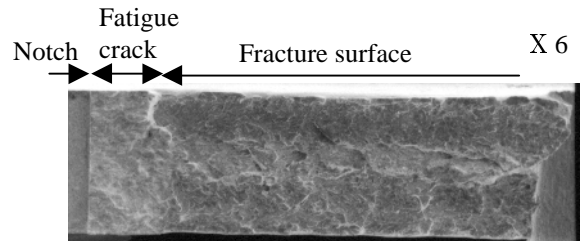Figure 8: Fracture surfaces tested at 4 K



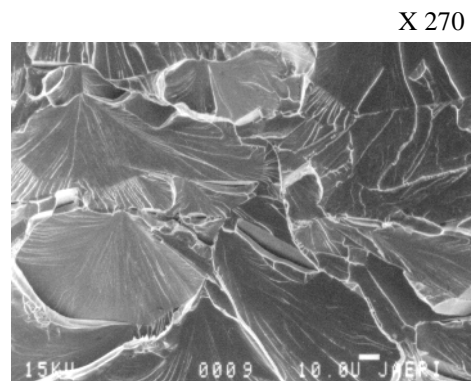Figure 9: Fracture surface of sample No.2 (Bulk Nb)


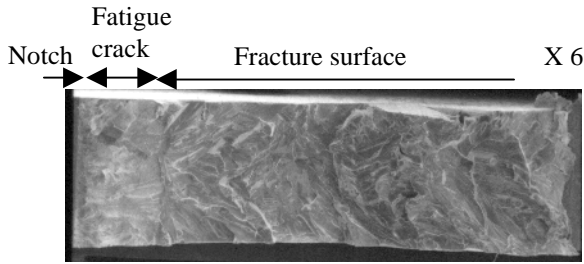
Figure 10: Cleavage fracture surface of sample No.2

Figure 11: Fracture surface of sample No3 (Weld bead)



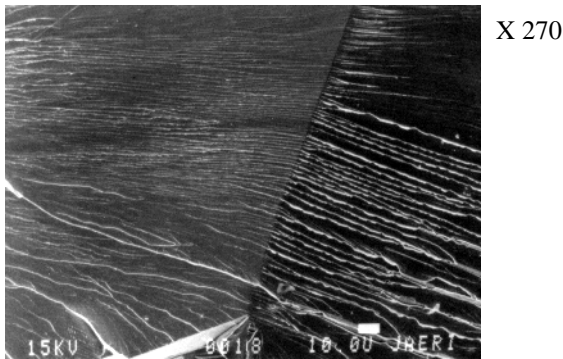Figure 12: Cleavage fracture surface of sample No.3



Figure 13: Cleavage fracture surface of sample No.3

Figure 14 to figure 16 show the metallographic sections of fractured surfaces of the sample No.2 , No.3 and No.4. Micro-cracks or microvoids along the slip bands, or at the intersection of , 1. grain boundary and slip bands, 2. crossed slip bands (compiled dislocations), were observed. The fracture initiates the microvoids and micro-cracks, and these defects finally cause the cleavage fracture.
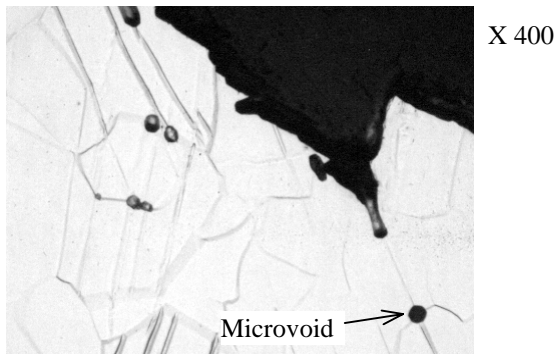


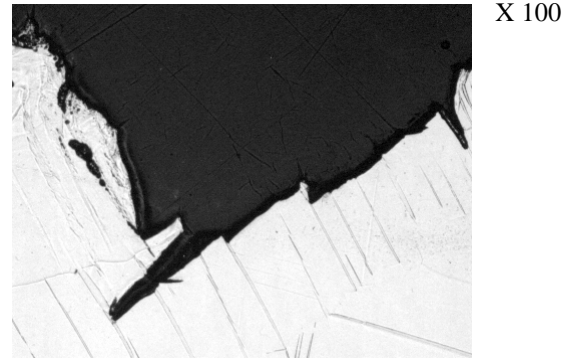Figure 14: Fracture with microvoids of sample No.2



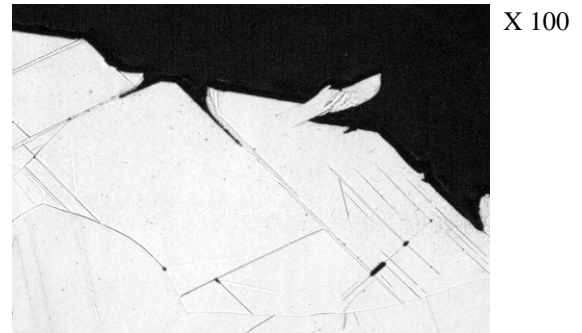Figure 15: Fracture with micro-cracks along slip bands of sample No.3



Figure 16: Fracture with micro-cracks and microvoids of sample No.4, revealing cracks along slip bands, microvoids at intersection of crossed slip bands

## 4 DISCUSSIONS

Here, we try to calculate the allowable pressure for crack propagation by using the obtained $K_Q$=33.5 MPa·m$^{1/2}$ of the weld bead. Our design stress level of a cavity is limited below 28.7 MPa (43 MPa/1.5, based on YS at RT) against the vacuum load.

### 4.1 Center-cracked model in a large sheet

The stress-intensity factor of this model shown in figure 17 is determined from Eq. 3.

$$K = \sigma\sqrt{\pi a} \qquad (3)$$

Table 4 shows the result of a calculation.

Table 4 : Allowable pressure

| Flaw size 2a | (mm) | 0.5 | 1 | 5 |
|---|---|---|---|---|
| Allowable pressure | atm | 41.7 | 29.5 | 13.2 |

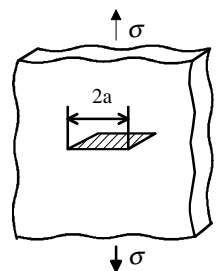| 10 | 20 | 40 | 80 | 160 | 320 |
|---|---|---|---|---|---|
| 9.3 | 6.6 | 4.7 | 3.3 | 2.3 | 1.6 |



Figure17:Center-cracked model

### 4.2 Thin-walled cylinder with a crack

The calculation model is shown in figure 18 with a crack of 1.5 mm depth in a thin-walled cylinder of 3 mm thickness.

Stress intensity factor of this model is given by Eq. 4,

and $K$ value of 5.57 MPa·m$^{1/2}$ is calculated under the pressure of 1 atm. Therefore, the critical pressure for crack propagation is 5.2 atm.

$$K = \sigma\sqrt{\pi a}\, F(a/W),\ a/W = \xi \quad (4)$$
$$F(\xi) \cong 1.12 - 0.231\xi + 10.55\xi^2$$
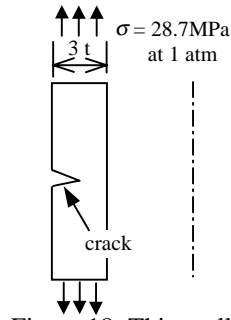$$- 21.72\xi^3 + 30.39\xi^4$$

Figure 18: Thin-walled cylinder model

## 5 CONCLUSIONS

1. The fracture toughness ($K_Q$) for bulk Nb at 4 K is 45.5 MPa·m$^{1/2}$, and 33.5 MPa·m$^{1/2}$ for weld bead.

2. The fracture of Nb at 4 K initiates the microvoids and micro-cracks, and finally these defects cause the cleavage fracture.

## 6 ACKNOWLEDGEMENT

## 7 REFERENCES

[1] A. L. Mincher, W. F. Sheely, "Effect of Structure and Purity on the Mechanical Properties of Columbium", Trans. of the Metallurgical Society of AIME, Vol 221, 1961, 19-24

[2] J. W. Cristian, B. C. Masters, "Low-temperature deformation of body-centred cubic metals", Proc. of the Royal Society of London, Vol A281, 223 (1964)

[3] M. G. Rao and P. Kneisel, "Thermal and Mechanical Properties of Electron Beam Welded and Heat-Treated Niobium for Tesla", Proc. of the 6th Workshop on RF-Superconductivity, CEBAF, 643-649 (1993)

[4] Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials, ASTM standard, E399-90

[5] Standard Test Method for J-integral Characterization of Fracture Toughness, ASTM standard, E1737-96

[6] Fracture mechanics toughness tests, Part 1. Method determination of K$_{IC}$, critical CTOD and critical J Values of metallic materials, BS7448-1991

## APPENDIX

*Referenced to the ASTM E399, E1737 and BS7448*

### 1. Pre-cracking Procedure (ASTM E399-90)

$K_{f(max)}/E \le 0.00032\,\mathrm{m}^{1/2}$
$K_{f(max)} \le 0.6\ (\sigma_{y1}/\sigma_{y2}) \times K_Q$
$-1 \le P_{min}/P_{max} \le 0.1$
$0.45W \le a \le 0.55W$, where:

$K_{f(max)}$ : max. stress intensity in the fatigue crack growth
$E$ : Young's modulus (MPa)
$\sigma_{y1}$ : yield stress in the fatigue pre-cracking (MPa)
$\sigma_{y2}$ : yield stress in the $K_{IC}$ test (MPa)

$K_Q$ : Calculated from 95% secant load (MPa·m$^{1/2}$)
$P_{min}/P_{max}$: a ratio of min. to max. load in the fatigue pre-cracking
$W$ = specimen width (cm)
$a$ = crack length (cm)

Example: Fatigue pre-cracking condition
0.2 % yield strength for lot 1 material
$\sigma_{y1}$: 44 MPa at RT, $\sigma_{y1}$: 444 MPa at 77 K
$\sigma_{y2}$: 431 MPa at 4 K
assuming $K_Q$ = 30 MPa·m$^{1/2}$
At room temperature,
$K_{f(max)}=0.6\ (\sigma_{y1}/\sigma_{y2}) \times K_Q = 1.84$ MPa·m$^{1/2}$
At LN$_2$ temperature (77 K),
$K_{f(max)}=19.10$ MPa·m$^{1/2}$

### 2. Calculation of $K_Q$ for the compact specimen in units of MPa·m$^{1/2}$ (ASTM E399-90)

$$K_Q = (P_Q/BW^{1/2}) \times \frac{(2+a/W)(0.886+4.64a/W-13.32a^2/W^2+14.72a^3/W^3-5.6a^4/W^4)}{(1-a/W)^{3/2}}$$

where:
$P_Q$ = load as determined, from 95 % secant load (kN)
$B$ = specimen thickness (cm)
$W$ = specimen width (cm)
$a$ = crack length (cm)

### 3. Crack length and thickness requirement (ASTM E399-90)

The crack length ($a$) and thickness ($B$) of the samples are needed to satisfy the plane-strain validity criteria require that

$$B\ \&\ a\ \ge\ 2.5\left(\frac{K_{IC}}{\sigma_{ys}}\right)^2$$

$B\ \&\ a$ : m, $\sigma_{ys}$ : MPa, $K_{IC}$ : MPa$\sqrt{m}$

### 4. Calculation of $J$-integral (ASTM E1737-96)

Calculations of $J$-integral are made from load and load-line displacement curves.
$$J = J_{el} + J_{pl}$$
where :
$J_{el}$ = elastic component of $J$
$J_{pl}$ = plastic cpmponent of $J$

$$J = \frac{K^2(1-v^2)}{E} + \frac{2A_{pl}}{Bb_0}$$

$A_{pl}$: area for J calculation
$b_0$: remaining ligament ($W$-$a$)

### 5. Calculation of load-line displacement (BS7448)

The load-line displacement $V_L$ can be calculated from the crack mouth open displacement $V_g$.
$$\frac{V_L}{Vg} = \frac{0.46W + 0.54a_0}{0.71W + 0.54a_0}$$